

PATENT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents
 United States Patent and Trademark
 Office
 Box PCT
 Washington, D.C.20231
 ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 26 July 2000 (26.07.00)	
International application No. PCT/GB99/04024	Applicant's or agent's file reference 285
International filing date (day/month/year) 01 December 1999 (01.12.99)	Priority date (day/month/year) 02 December 1998 (02.12.98)
Applicant KATHIRGAMANATHAN, Poopathy	

1. The designated Office is hereby notified of its election made:



in the demand filed with the International Preliminary Examining Authority on:

27 June 2000 (27.06.00)



in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was

was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer Zakaria EL KHODARY Telephone No.: (41-22) 338.83.38
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PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference 285	FOR FURTHER ACTION <small>see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.</small>	
International application No. PCT/GB 99/ 04024	International filing date (day/month/year) 01/12/1999	(Earliest) Priority Date (day/month/year) 02/12/1998
Applicant SOUTH BANK UNIVERSITY ENTERPRISES LTD et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 4 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

a. With regard to the language, the International search was carried out on the basis of the International application in the language in which it was filed, unless otherwise indicated under this item.

☐ the International search was carried out on the basis of a translation of the International application furnished to this Authority (Rule 23.1(b)).

b. With regard to any nucleotide and/or amino acid sequence disclosed in the International application, the International search was carried out on the basis of the sequence listing :

☐ contained in the International application in written form.

☐ filed together with the International application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the International application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ Certain claims were found unsearchable (See Box I).

3. ☐ Unity of invention is lacking (see Box II).

4. With regard to the title,

☐ the text is approved as submitted by the applicant.

☒ the text has been established by this Authority to read as follows:

ELECTROLUMINESCENT QUINOLATES

5. With regard to the abstract,

☐ the text is approved as submitted by the applicant.

☒ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this International search report, submit comments to this Authority.

6. The figure of the drawings to be published with the abstract is Figure No.

☐ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

☒ None of the figures.

Box III TEXT OF THE ABSTRACT (Continuation of item 5 of the first sheet)

Lithium quinolate is an electroluminescent material which emits light in the blue region of the spectrum.

PC B 99/04024

IPC 7 C09K11/06 H05B33/14 C07D215/30

IPC 7 C09K H05B C07D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

Y Patent family members are listed in annex.

"&" document member of the same patent family

Shade, M

INTERNATIONAL SEARCH REPORT

International Application No

PO 8 99/04024

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 755 999 A (CHEN CHIN H ET AL) 26 May 1998 (1998-05-26) column 35, line 19 ----	1,9-22
X	EP 0 569 827 A (IDEMITSU KOSAN CO) 18 November 1993 (1993-11-18) page 19, line 31 ----	1,9,10
A	PATENT ABSTRACTS OF JAPAN vol. 018, no. 462 (C-1243), 29 August 1994 (1994-08-29) & JP 06 145146 A (CHISSO CORP), 24 May 1994 (1994-05-24) abstract -----	1,9,10

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

P B 99/04024

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
EP 0936844	A	18-08-1999	JP 11233262 A	27-08-1999
WO 9802018	A	15-01-1998	DE 19627070 A	08-01-1998
			AU 3692497 A	02-02-1998
			EP 0910930 A	28-04-1999
US 5755999	A	26-05-1998	EP 0878529 A	18-11-1998
			JP 10330744 A	15-12-1998
EP 0569827	A	18-11-1993	JP 6313168 A	08-11-1994
			US 5516577 A	14-05-1996
JP 06145146	A	24-05-1994	NONE	

REC'D 12 MAR 2001

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 285	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/GB99/04024	International filing date (day/month/year) 01/12/1999	Priority date (day/month/year) 02/12/1998
International Patent Classification (IPC) or national classification and IPC C09K11/06		
Applicant SOUTH BANK UNIVERSITY ENTERPRISES LTD et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.


2. This REPORT consists of a total of 8 sheets, including this cover sheet.

- ☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☒ Certain documents cited
- VII ☒ Certain defects in the international application
- VIII ☒ Certain observations on the international application

Date of submission of the demand 27/06/2000	Date of completion of this report 08.03.2001
Name and mailing address of the international preliminary examining authority:  European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016	Authorized officer Shade, M Telephone No. +31 70 340 2332



INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB99/04024

I. Basis of the report

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

Description, pages:

1-10 as originally filed

Claims, No.:

1-22 as originally filed

Drawings, sheets:

1/7-7/7 as originally filed

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB99/04024

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	5, 7, 9-22
	No:	Claims	1, 2, 3, 4, 6
Inventive step (IS)	Yes:	Claims	
	No:	Claims	1-7, 9-22
Industrial applicability (IA)	Yes:	Claims	1-22
	No:	Claims	

2. Citations and explanations
see separate sheet

VI. Certain documents cited

1. Certain published documents (Rule 70.10)

and / or

2. Non-written disclosures (Rule 70.9)

see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:
see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

1) Reference is made to the following documents:

D1: J. Organomet. Chem. (1981), 209(1), 17-23 (0000), , -
D2: EP-A-0936844
D3: WO-A-9802018
D4: US-A-5755999
D5: EP-A-0569827
D6: JP(A) 06145146

2) Re Item V

Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Claims 1 to 22 do not satisfy the criteria of Articles 33(2) and (3) PCT.

2.1) Lithium quinolate and its synthesis from an alkyl lithium compound, in this case butyl lithium, and 8-hydroxyquinoline in a solvent is disclosed in document D1, see page 21, paragraph 4. The method of preparation of lithium quinolate disclosed in D1 involves the formation of a precipitate which, it can be assumed, will be deposited on the surface of the vessel. Therefore the subject-matter of claims 1, 2, 3, 4 and 6 does not satisfy the criteria of Article 33(2) PCT. The compound lithium quinolate is also disclosed in documents D3, page 28, line 13, D4, column 35, line 19 and D5, page 19, line 31.

2.2) The subject-matter of present claim 7, directed to the synthesis of lithium quinolate using 8-hydroxyquinoline and a specific lithium alkoxide is not inventive as the substitution of a lithium alkoxide for the lithium alkyl of D1 would be obvious to one skilled in the art, the specific lithium alkoxides mentioned in claim 7 are those commonly used in synthetic organic chemistry. The subject-matter of claim 7 does not satisfy the criteria of Article 33(3) PCT.

2.3) The subject-matter of claim 5 is not inventive since it would be obvious to a person skilled in the art to carry out the reaction carried out in bulk solution in D1, see paragraph 2.1) above, in a film of solution on a substrate in order to obtain a precipitate deposited over the substrate. The subject-matter of claim 5 does not satisfy the criteria

of Article 33(3) PCT.

2.4) With reference to the subject-matter of claims 9 and 10 document D3 discloses an electroluminescent device comprising an anode and a cathode, at least one of which is transparent, a hole-transporting compound and a luminescent compound, the luminescent compound is selected from a range of quinolate complexes including lithium quinolate, see D3, claims 1, 6 and 9.

The subject-matter of present claims 9 and 10 consists in a structure incorporating a layer of luminescent material and a means to pass electric current through that material and an electroluminescent device comprising a luminescent material in which the luminescent material has been selected from the range of luminescent materials described in document D3, see claims 1, 6 and 9. Such a selection can only be regarded as inventive, if the resultant structure or electroluminescent device presents unexpected effects or properties in relation to the rest of the range. However, no such effects or properties are indicated in the application. Hence, no inventive step is present in the subject-matter of claims 9 and 10. Claims 9 and 10 do not meet the requirements of Article 33(3) PCT

2.5) Dependent claims 11 to 22 do not contain any features which, in combination with the features of any claim to which they refer, meet the requirements of the PCT in respect of novelty or inventive step, the reasons being as follows:

- i) regarding the subject-matter of present claim 11, materials which may be used as the transparent substrate of electroluminescent devices are well known in the art, see D3, page 29, lines 16 to 22.
- ii) regarding the subject-matter of present claims 12, 13, 17 to 22, the different arrangements which may be used in electroluminescent devices and the specific materials mentioned by the applicant for use as hole transport materials and electron transport materials are well known in the art, see D4, column 2, line 64 to column 4, line 11, column 5, line 30 to column 7, line 30 and column 34, line 44 to column 35, line 20.
- iii) regarding the subject-matter of claim 14, the use of dyes to modify the colour of the emitted light in electroluminescent devices is well known in the art, see D4, column 7,

lines 31 to 48.

iv) regarding the subject-matter of present claims 15 and 16 the use of polymeric binders in the emitting layers of electroluminescent devices and the relative quantity of active component to binder which may be used is known in the art, see D3, page 27, lines 4 to 15.

2.6) The subject-matter of claims 1-22 satisfies the criteria of Article 33(1) and (4) PCT, said subject-matter having industrial application in the field of electroluminescent devices.

3) Re Item VI

Certain documents cited

Certain published documents (Rule 70.10)

Application No	EP936844
Publication date (day/month/year)	18/8/1999
Filing date (day/month/year)	12/02/1999
Priority date (day/month/year)	17/02/1998

Document EP936844, D2, discloses the subject-matter of present claims 1, 9, 10 and 11, see D2, Example 1 which discloses an electroluminescent device comprising an ITO anode, a layer of hole transporting tertiary amine, a light emitting layer of aluminium trisquinoline, an electron transporting layer of lithium quinolate and a cathode.

4) Re Item VII

Certain defects in the international application

4.1) Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in the documents D1, D3, D4 and D5 is not mentioned in the description, nor are these documents identified therein.

5) Re Item VIII

Certain observations on the international application

5.1) In the description there are a number of embodiments of the invention which do not fall within the scope of the claims. This inconsistency between the claims and the description leads to doubt concerning the matter for which protection is sought, thereby rendering the claims unclear (Article 6 PCT). The embodiments concerned are as follows:

- i) Page 1, lines 28 and 29, page 2, lines 12 to 21, indicate that methods of making metal quinolates are embodiments of the invention, only a method of making lithium quinolate is claimed.
- ii) Page 2, line 27 to page 3, line 5 indicates that substituted lithium quinolates are also embodiments of the invention whereas only lithium quinolate is claimed. Substituted lithium quinolates are disclosed in D6.
- iii) Examples 3 to 7 on pages 6 and 7, indicate that other metal quinolates are embodiments of the invention whereas only lithium quinolate is claimed.

5.2) With reference to the numbering of the claims, contrary to Rule 6.1(b) PCT there is no claim 8 and contrary to Rule 6.4(a) PCT claim 16 is dependent on itself.

5.3) The molecular formulae given in examples 3 to 7 are incorrect.

5.4) The unit torr employed on page 9 is not additionally expressed in terms of the units stipulated by Rule 10.1(a) PCT.

5.5) The dependency of claim 17 is such that it renders its subject-matter unclear, this leads to a lack of clarity of the claims as a whole, contrary to Article 6 PCT. Claim 17 refers to an electroluminescent device comprising a layer which is a mixture of lithium quinolate and hole transporting material, it is dependent on claim 12 which is directed to an electroluminescent device comprising separate hole transporting and emitting layers. It is not clear whether the device of claim 17 comprises both a hole transporting layer and a further layer which is a mixture of hole transporting material and lithium quinolate or a single layer comprising a mixture of hole transporting material and lithium

quinolate.

5.6) The subject-matter of claim 7 is not clear, claim 7 refers to the metal alkoxide of any of claims 2 to 5, whereas claims 2 to 5 refer only to lithium alkoxide.

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

COHEN, Alan, Nicol
2 Grove Place
Tatsfield, Westerham
Kent TN16 2BB
GRANDE BRETAGNE

PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT (PCT Rule 71.1)

Date of mailing (day/month/year)	08.03.2001
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Applicant's or agent's file reference 285	IMPORTANT NOTIFICATION
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International application No. PCT/GB99/04024	International filing date (day/month/year) 01/12/1999	Priority date (day/month/year) 02/12/1998
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Applicant SOUTH BANK UNIVERSITY ENTERPRISES LTD et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.
4. **REMINDER**

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/	Authorized officer
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PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 285	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/PEA/416)	
International application No. PCT/GB99/04024	International filing date (day/month/year) 01/12/1999	Priority date (day/month/year) 02/12/1998
International Patent Classification (IPC) or national classification and IPC C09K11/06		
Applicant SOUTH BANK UNIVERSITY ENTERPRISES LTD et al.		
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 9 sheets, including this cover sheet.</p> <p><input type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of sheets.</p>		
<p>3. This report contains indications relating to the following items:</p> <ul style="list-style-type: none"> I <input checked="" type="checkbox"/> Basis of the report II <input type="checkbox"/> Priority III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability IV <input type="checkbox"/> Lack of unity of invention V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement VI <input checked="" type="checkbox"/> Certain documents cited VII <input checked="" type="checkbox"/> Certain defects in the international application VIII <input checked="" type="checkbox"/> Certain observations on the international application 		
Date of submission of the demand 27/06/2000	Date of completion of this report 08.03.2001	
Name and mailing address of the international preliminary examining authority:  European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016	Authorized officer Shade, M Telephone No. +31 70 340 2332	



**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB99/04024

I. Basis of the report

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

Description, pages:

1-10 as originally filed

Claims, No.:

1-22 as originally filed

Drawings, sheets:

1/7-7/7 as originally filed

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB99/04024

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):
(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims 5, 7, 9-22
	No:	Claims 1, 2, 3, 4, 6
Inventive step (IS)	Yes:	Claims
	No:	Claims 1-7, 9-22
Industrial applicability (IA)	Yes:	Claims 1-22
	No:	Claims

2. Citations and explanations **see separate sheet**

VI. Certain documents cited

1. Certain published documents (Rule 70.10)

and / or

2. Non-written disclosures (Rule 70.9)

see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:
see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/GB99/04024

1) Reference is made to the following documents:

D1:J. Organomet. Chem. (1981), 209(1), 17-23 (0000), , -

D2:EP-A-0936844

D3:WO-A-9802018

D4:US-A-5755999

D5:EP-A-0569827

D6:JP(A) 06145146

2) Re Item V

Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Claims 1 to 22 do not satisfy the criteria of Articles 33(2) and (3) PCT.

2.1) Lithium quinolate and its synthesis from an alkyl lithium compound, in this case butyl lithium, and 8-hydroxyquinoline in a solvent is disclosed in document D1, see page 21, paragraph 4. The method of preparation of lithium quinolate disclosed in D1 involves the formation of a precipitate which, it can be assumed, will be deposited on the surface of the vessel. Therefore the subject-matter of claims 1, 2, 3, 4 and 6 does not satisfy the criteria of Article 33(2) PCT. The compound lithium quinolate is also disclosed in documents D3, page 28, line 13, D4, column 35, line 19 and D5, page 19, line 31.

2.2) The subject-matter of present claim 7, directed to the synthesis of lithium quinolate using 8-hydroxyquinoline and a specific lithium alkoxide is not inventive as the substitution of a lithium alkoxide for the lithium alkyl of D1 would be obvious to one skilled in the art, the specific lithium alkoxides mentioned in claim 7 are those commonly used in synthetic organic chemistry. The subject-matter of claim 7 does not satisfy the criteria of Article 33(3) PCT.

2.3) The subject-matter of claim 5 is not inventive since it would be obvious to a person skilled in the art to carry out the reaction carried out in bulk solution in D1, see paragraph 2.1) above, in a film of solution on a substrate in order to obtain a precipitate deposited over the substrate. The subject-matter of claim 5 does not satisfy the criteria

of Article 33(3) PCT.

2.4) With reference to the subject-matter of claims 9 and 10 document D3 discloses an electroluminescent device comprising an anode and a cathode, at least one of which is transparent, a hole-transporting compound and a luminescent compound, the luminescent compound is selected from a range of quinolate complexes including lithium quinolate, see D3, claims 1, 6 and 9.

The subject-matter of present claims 9 and 10 consists in a structure incorporating a layer of luminescent material and a means to pass electric current through that material and an electroluminescent device comprising a luminescent material in which the luminescent material has been selected from the range of luminescent materials described in document D3, see claims 1, 6 and 9. Such a selection can only be regarded as inventive, if the resultant structure or electroluminescent device presents unexpected effects or properties in relation to the rest of the range. However, no such effects or properties are indicated in the application. Hence, no inventive step is present in the subject-matter of claims 9 and 10. Claims 9 and 10 do not meet the requirements of Article 33(3) PCT

2.5) Dependent claims 11 to 22 do not contain any features which, in combination with the features of any claim to which they refer, meet the requirements of the PCT in respect of novelty or inventive step, the reasons being as follows:

i) regarding the subject-matter of present claim 11, materials which may be used as the transparent substrate of electroluminescent devices are well known in the art, see D3, page 29, lines 16 to 22.

ii) regarding the subject-matter of present claims 12, 13, 17 to 22, the different arrangements which may be used in electroluminescent devices and the specific materials mentioned by the applicant for use as hole transport materials and electron transport materials are well known in the art, see D4, column 2, line 64 to column 4, line 11, column 5, line 30 to column 7, line 30 and column 34, line 44 to column 35, line 20.

iii) regarding the subject-matter of claim 14, the use of dyes to modify the colour of the emitted light in electroluminescent devices is well known in the art, see D4, column 7,

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/GB99/04024

lines 31 to 48.

iv) regarding the subject-matter of present claims 15 and 16 the use of polymeric binders in the emitting layers of electroluminescent devices and the relative quantity of active component to binder which may be used is known in the art, see D3, page 27, lines 4 to 15.

2.6) The subject-matter of claims 1-22 satisfies the criteria of Article 33(1) and (4) PCT, said subject-matter having industrial application in the field of electroluminescent devices.

3) Re Item VI

Certain documents cited

Certain published documents (Rule 70.10)

Application No	EP936844
Publication date (day/month/year)	18/8/1999
Filing date (day/month/year)	12/02/1999
Priority date (day/month/year)	17/02/1998

Document EP936844, D2, discloses the subject-matter of present claims 1, 9, 10 and 11, see D2, Example 1 which discloses an electroluminescent device comprising an ITO anode, a layer of hole transporting tertiary amine, a light emitting layer of aluminium trisquinoline, an electron transporting layer of lithium quinolate and a cathode.

4) Re Item VII

Certain defects in the international application

4.1) Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in the documents D1, D3, D4 and D5 is not mentioned in the description, nor are these documents identified therein.

5) Re Item VIII

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/GB99/04024

Certain observations on the international application

5.1) In the description there are a number of embodiments of the invention which do not fall within the scope of the claims. This inconsistency between the claims and the description leads to doubt concerning the matter for which protection is sought, thereby rendering the claims unclear (Article 6 PCT). The embodiments concerned are as follows:

- i) Page 1, lines 28 and 29, page 2, lines 12 to 21, indicate that methods of making metal quinolates are embodiments of the invention, only a method of making lithium quinolate is claimed.
- ii) Page 2, line 27 to page 3, line 5 indicates that substituted lithium quinolates are also embodiments of the invention whereas only lithium quinolate is claimed. Substituted lithium quinolates are disclosed in D6.
- iii) Examples 3 to 7 on pages 6 and 7, indicate that other metal quinolates are embodiments of the invention whereas only lithium quinolate is claimed.

5.2) With reference to the numbering of the claims, contrary to Rule 6.1(b) PCT there is no claim 8 and contrary to Rule 6.4(a) PCT claim 16 is dependent on itself.

5.3) The molecular formulae given in examples 3 to 7 are incorrect.

5.4) The unit torr employed on page 9 is not additionally expressed in terms of the units stipulated by Rule 10.1(a) PCT.

5.5) The dependency of claim 17 is such that it renders its subject-matter unclear, this leads to a lack of clarity of the claims as a whole, contrary to Article 6 PCT. Claim 17 refers to an electroluminescent device comprising a layer which is a mixture of lithium quinolate and hole transporting material, it is dependent on claim 12 which is directed to an electroluminescent device comprising separate hole transporting and emitting layers. It is not clear whether the device of claim 17 comprises both a hole transporting layer and a further layer which is a mixture of hole transporting material and lithium quinolate or a single layer comprising a mixture of hole transporting material and lithium

INTERNATIONAL PRELIMINARY

International application No. PCT/GB99/04024

EXAMINATION REPORT - SEPARATE SHEET

quinolate.

5.6) The subject-matter of claim 7 is not clear, claim 7 refers to the metal alkoxide of any of claims 2 to 5, whereas claims 2 to 5 refer only to lithium alkoxide.

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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁷ : C09K 11/06, H05B 33/14, C07D 215/30		A1	(11) International Publication Number: WO 00/32717
			(43) International Publication Date: 8 June 2000 (08.06.00)
(21) International Application Number: PCT/GB99/04024 (22) International Filing Date: 1 December 1999 (01.12.99) (30) Priority Data: 9826406.2 2 December 1998 (02.12.98) GB (71) Applicant (for all designated States except US): SOUTH BANK UNIVERSITY ENTERPRISES LTD. [GB/GB]; 103 Borough Road, London SE1 0AA (GB). (72) Inventor; and (75) Inventor/Applicant (for US only): KATHIRGAMANATHAN, Poopathy [GB/GB]; 14 Sandhurst Avenue, North Harrow, Middlesex HA2 7AP (GB). (74) Agent: COHEN, Alan, Nicol; 2 Grove Place, Tatsfield, Westerham, Kent TN16 2BB (GB).			(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: ELECTROLUMINESCENT QUINOLATES			
(57) Abstract Lithium quinolate is an electroluminescent material which emits light in the blue region of the spectrum.			

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ELECTROLUMINESCENT QUINOLATES

The present invention relates to novel photoluminescent and electroluminescent materials.

5

Aluminium quinolate is a known photoluminescent and electroluminescent material and emits light in the red area of the spectrum. In order to obtain light of a different wavelength dopants and/or dyes have been added to the aluminium quinolate. Structures have also been made with a layer containing dyes in contact with the aluminium quinolate layer but aluminium quinolate and structures based on aluminium quinolate have a relatively low efficiency.

10

In an article by Takeo Wakimoto et al in Applied Surface Science 113/114(1997) 698-704 electroluminescent cells are disclosed in which aluminium quinolate is used as the emitter and which is doped by quinacrodine derivatives which are fluorescent dyes to change the colour of the emitted light.

15

Electroluminescent devices can be made as described in an article by K. Nagayama et al in the Jpn. Journal of Applied Physics vol. 36 pps. 1555-1557.

20

The obtaining of blue light in an electroluminescent material is required to enable the complete range of colours to be obtained in devices incorporating such materials.

We have now obtained a novel electroluminescent material which emits blue light.

25

One aspect of the invention is lithium quinolate.

Another aspect of the invention is a method of making a metal quinolate which comprises reacting a metal alkyl or a metal alkoxide with 8-hydroxy quinoline.

30

- 2 -

A further aspect of the invention is the provision of a structure which incorporates a layer of lithium quinolate and a means to pass an electric current through the lithium quinolate layer.

- 5 Although some metal quinolates are known hitherto lithium quinolate has not been made and it was surprising that it was photoluminescent and electroluminescent in the blue spectrum.

- 10 Aluminium and other known metal quinolates are made by the reaction of a metal salt with 8-hydroxyquinoline.

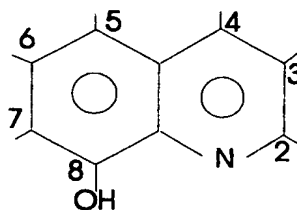
- 15 In the method of the invention the metal alkyl or alkoxide is preferably reacted in the liquid phase with the 8-hydroxyquinoline. The metal compound can be dissolved in an inert solvent added to the 8-hydroxyquinoline. The metal quinolate can be separated by evaporation or when a film of the metal quinolate is required, by deposition onto a suitable substrate.

- 20 The preferred alkyls are ethyl, propyl and butyl with n-butyl being particularly preferred. With metal alkoxides the preferred alkoxides are ethoxide, propoxides and butoxides. The method is particularly suitable for the preparation of group I, II and III metals such as lithium, sodium potassium, zinc, cadmium and aluminium alkoxides.

- 25 Lithium quinolate can be synthesised by the reaction, in an inert solvent, e.g. acetonitrile, of 8-hydroxyquinoline with a lithium alkyl e.g. n-butyl lithium. The lithium quinolate is an off white or white solid at room temperature.

As well as the lithium salt of 8-hydroxyquinoline, the term quinolate in this specification includes salts of substituted 8-hydroxyquinoline

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where the substituents are the same or different in the 2, 3, 4, 5, 6 and 7 positions and are selected from alky, alkoxy, aryl, aryloxy, sulphonic acids, esters, carboxylic acids, amino and amido groups or are aromatic, polycyclic or heterocyclic groups.

Alkali metal alkyls are difficult compounds to handle practically as they are highly reactive and can catch fire spontaneously in air. For this reason they would not normally be chosen as reactants.

An electroluminescent device comprises a conductive substrate which acts as the anode, a layer of the electroluminescent material and a metal contact connected to the electroluminescent layer which acts as the cathode. When a current is passed through the electroluminescent layer, the layer emits light.

Preferably the electroluminescent devices of the invention comprise a transparent substrate, which is a conductive glass or plastic material which acts as the anode, preferred substrates are conductive glasses such as indium tin oxide coated glass, but any glass which is conductive or has a conductive layer can be used. Conductive polymers and conductive polymer coated glass or plastics materials can also be used as the substrate. The lithium quinolate can be deposited on the substrate directly by evaporation from a solution in an organic solvent. Any solvent which dissolves the lithium quinolate can be used e.g. acetonitrile.

A preferred method of forming a metal quinolate film e.g. useful in

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electroluminescent devices comprises forming the metal quinolate in situ by sequential dip coating the substrate with the film e.g. the substrate is dipped or otherwise coated with a solution of the metal alkyl or alkoxide to form a film on the surface and then dipped or otherwise coated with 8-hydroxyquinoline or substituted

5 8-hydroxyquinoline and the metal quinolate film is formed on the substrate surface.

For example to form a film of lithium quinolate the film or layer of lithium quinolate is deposited by in situ dip coating i.e. the substrate, such as a glass slide, is dipped into or otherwise contacted with a solution of an alkyl lithium e.g. n-butyl lithium and

10 then immersed in or contacted with a solution of hydroxyquinoline, a layer of lithium quinolate is then formed on the substrate.

Alternatively the material can be deposited by spin coating or by vacuum deposition from the solid state e.g. by sputtering or any other conventional method can be used.

15 To form an electroluminescent device incorporating lithium quinolate as the emissive layer there can be a hole transporting layer deposited on the transparent substrate and the lithium quinolate is deposited on the hole transporting layer. The hole transporting layer serves to transport; holes and to block the electrons, thus

20 preventing electrons from moving into the electrode without recombining with holes. The recombination of carriers therefore mainly takes place in the emitter layer.

Hole transporting layers are used in polymer electroluminescent devices and any of the known hole transporting materials in film form can be used.

25 The hole transporting layer can be made of a film of an aromatic amine complex such as poly(vinylcarbazole), N,N'-diphenyl-N,N'-bis (3-methylphenyl)-I,I'-biphenyl -4,4'-diamine (TPD), polyaniline etc.

30 Optionally dyes such as fluorescent laser dyes, luminescent laser dyes can be included

to modify the colour spectrum of the emitted light.

The lithium quinolate can be mixed with a polymeric material such as a polyolefin e.g. polyethylene, polypropylene etc. and preferably polystyrene. Preferred amounts
5 of active material in the mixture is from 95% to 5% by weight of active material and more preferably 25 to 20% by weight.

The hole transporting material can optionally be mixed with the lithium quinolate in a ratio of 5-95% of the lithium quinolate to 95 to 5% of the hole transporting
10 compound. In another embodiment of the invention there is a layer of an electron injecting material between the cathode and the lithium quinolate layer, this electron injecting layer is preferably a metal complex such as a different metal quinolate e.g. an aluminium quinolate which will transport electrons when an electric current is passed through it. Alternatively the electron injecting material can be mixed with the
15 lithium quinolate and co-deposited with it.

In another embodiment of the invention there is a layer of an electron transporting material between the cathode and the lithium quinolate layer, this electron transporting layer is preferably a metal complex such as a metal quinolate e.g. an
20 aluminium quinolate which will transport electrons when an electric current is passed through it. Alternatively the electron transporting material can be mixed with the lithium quinolate and co-deposited with it.

Optionally dyes such as fluorescent laser dyes, luminescent laser dyes can be included
25 to modify the colour spectrum of the emitted light and also enhance the photoluminescent and electroluminescent efficiencies.

In a preferred structure there is a substrate formed of a transparent conductive material which is the anode on which is successively deposited a hole transportation
30 layer, the lithium quinolate layer and an electron transporting layer which is

connected to the anode. The anode can be any low work function metal e.g. aluminium, calcium, lithium, silver/magnesium alloys etc.

The invention is further described with reference to the examples.

5

Example 1 Lithium 8-hydroxyquinolate $\text{Li}(\text{C}_9\text{H}_6\text{ON})$

2.32g (0.016 mole) of 8-hydroxyquinoline was dissolved in acetonitrile and 10ml of 1.6M n-butyl lithium (0.016 mole) was added. The solution was stirred at room temperature for one hour and an off white precipitate filtered off . The precipitate was washed with water followed by acetonitrile and dried in vacuo. The solid was shown to be lithium quinolate.

10

Example 2 Lithium 8-hydroxyquinolate $\text{Li}(\text{C}_9\text{H}_6\text{ON})$

15

A glass slide (Spectrosil UV grade) was dipped into a solution of n-butyl lithium in acetonitrile for four seconds and then immersed in a solution of 8 - hydroxyquinoline for four seconds. A thin layer of lithium quinolate was easily seen on the glass.

20

Example 3 Magnesium 8-hydroxyquinolate $\text{Mg}(\text{C}_9\text{H}_6\text{ON})_2$

25

8-Hydroxyquinoline (5.0 -g; 0.0345 mole) was dissolved in 2N acetic acid (150 ml) by heating at 70 - 80°C. Magnesium sulphate (2.5 g; 0.020 mole) was dissolved in water (100 ml) heated to 60°C and basified with ammonia. Oxine solution was added to the mechanically stirred, basified magnesium sulphate solution at 60°C and excess ammonia added until the pH of the solution was 9.5. The yellow precipitate was digested at 60°C for a further 10 minutes, cooled and filtered under suction, washed with dilute ammonia and dried in vacuo at 100°C for several hours. Yield 5.06. g

30

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Example 4 Zinc 8-hydroxyquinolate $\text{Zn}(\text{C}_2\text{H}_6\text{ON})_2$

The above procedure was employed using 8-hydroxyquinoline (5.0 g; 0.0345 mole) and zinc chloride (2.8 g; 0.020 mole). The yellow precipitate was filtered, washed
5 with dilute ammonia and dried in vacuo at 75°C for 6 hours. Yield 6.48 g

Example 5 Calcium 8-hydroxyquinolate $\text{Ca}(\text{C}_2\text{H}_6\text{ON})_2$

Using similar procedure with 8-hydroxyquinoline (5.0 g; 0.0345 mole) and
10 calcium chloride (3.8 g; 0.034 mole), calcium 8-hydroxyquinolate was obtained as a yellow powder 5.60 g yield.

Example 6 Sodium 8-hydroxyquinolate $\text{Na}(\text{C}_2\text{H}_6\text{ON})$

15 8-Hydroxyquinoline (5.0 g; 0.0345 mole) was dissolved in 2 % sodium hydroxide solution (100 ml) and heated to 60°C. The solution was stirred at this temperature for 30 minutes and the homogeneous solution was cooled to room temperature. No solid was separated out. Therefore the solution was concentrated in a rotary evaporator and the concentrated solution was cooled to give a
20 pale yellow solid. The solid was filtered under suction washed with small amounts of sodium hydroxide solution and dried in vacuo at 80°C for several hours. The sodium 8-hydroxyquinolate is soluble in water. Yield 3.6 g.

Example 7 Potassium 8-hydroxyquinolate $\text{K}(\text{C}_2\text{H}_6\text{ON})$

25

Potassium 8-hydroxyquinolate was also made from 8-hydroxyquinoline (2.0 g; 0.0138 mole) in dry tetrahydrofuran (50 ml) and potassium tert-butoxide (2.32 g; 0.021 mole). The solution was heated to become homogeneous and cooled to room temperature to give a yellow solid yield 2.2 g.

30

The photoluminescent efficiency and maximum wavelength of the PL emission of the lithium quinolate was measured and compared with other metal quinolates and the results shown in Table 1. Photoluminescence was excited using 325nm line of Liconix 4207 NB, He/Cd laser. The laser power incident at the sample (0.3mWcm^{-2}) was measured by a Liconix 55PM laser power meter. The radiance calibration was carried out using Bentham radiance standard (Bentham SRS8, Lamp current 4,000A), calibrated by National Physical laboratories, England. The PL studies were carried out on samples or films. The Spectra are attached as Figs. 2 to 7.

10

Table 1

				Absolute Photoluminescent Efficiency %
	Complex	CIE x,y	λ_{max} (PL)/nm	η_{PL}
15	Liq	0.17, 0.23	465	48
	Naq	0.19, 0.31	484	32
	Kq	0.19, 0.33	485	36
	Baq ₂	0.16, 0.29	479	7
	Caq ₂	0.21, 0.37	482	24
20	Mgq ₂	0.22, 0.46	500	43
	Znq ₂	0.26, 0.51	518	
	Alq ₃	0.32, 0.56	522	27

Example 8

25

An electroluminescent device of structure shown in fig. 1 was fabricated using aluminium quinolate and lithium quinolate as the electroluminescent layer and the electroluminescent properties measured. Referring to fig. 1 (2) is an ITO layer, (4) is a TPD layer (hole transporting layer) (60nm), (1) is the lithium quinolate layer (5) is an aluminium quinolate layer and (3) is aluminium (900nm).

30

1. Device Fabrication

5 An ITO coated glass piece ($1 \times 1\text{cm}^2$ cut from large sheets purchased from Balzers, Switzerland) had a portion etched out with concentrated hydrochloric acid to remove the ITO and was cleaned and placed on a spin coater (CPS 10 BM, Semitec, Germany) and spun at 2000 rpm for 30 seconds, during which time the solution of the electroluminescent compound was dropped onto it dropwise by a pipette.

10 Alternatively the electroluminescent compound was vacuum evaporated onto the ITO coated glass piece by placing the substrate in a vacuum coater and evaporating the electroluminescent compound at 10^{-5} to 10^{-6} torr onto the substrate.

15 The organic coating on the portion which had been etched with the concentrated hydrochloric acid was wiped with a cotton bud.

20 The coated electrodes were stored in a vacuum desiccator over calcium sulphate until they were loaded into a vacuum coater (Edwards, 10^{-6} torr) and aluminium top contacts made. The active area of the LED's was 0.08 cm^2 by 0.1 cm^2 the devices were then kept in a vacuum desiccator until the electroluminescence studies were performed.

25 The ITO electrode was always connected to the positive terminal. The current vs. voltage studies were carried out on a computer controlled Keithly 2400 source meter.

Electroluminescence spectra were recorded by means of a computer controlled charge coupled device on Insta Spec photodiode array system model 77112 (Oriel Co. Surrey, England)

30 The spectra are shown in the drawings.

In the spectra:-

Fig. 2 shows the PL of lithium 8-hydroxyquinolate of Example 1 and the quinolates of Examples 6 and 7.

- 5** Fig. 3 shows the PL of quinolates of Examples 3 and 5 and that of barium quinolate made by the same method.

Fig. 4 shows the PL of zinc quinolate of Example 4

Fig. 5 shows the PL of commercially available aluminium quinolate and

Fig. 6 shows the UV-VIS, PL and EL of lithium quinolate and

- 10** Fig. 7 shows the spectra of lithium quinolate of Example 2

Claims

1. Lithium quinolate.
- 5 2. A method of making lithium quinolate which comprises reacting a lithium alkyl or alkoxide with 8-hydroxy quinoline or substituted 8-hydroxy quinoline.
3. A method of making lithium quinolate as claimed in claim 2 in which a lithium alkyl or alkoxide is reacted with the 8-hydroxyquinoline in the liquid phase.
- 10 4. A method of making lithium quinolate as claimed in claim 3 in which a film of lithium quinolate is deposited onto a substrate.
5. A method as claimed in claim 4 in which the substrate is dipped or otherwise coated with a solution of the lithium alkyl or alkoxide to form a film on the surface and then dipped or otherwise coated with 8-hydroxyquinoline and the lithium quinolate film is formed on the substrate surface.
- 15 6. A method as claimed in any one of claims 2 to 5 in which the alkyl is ethyl, propyl or butyl.
- 20 7. A method as claimed in any one of claims 2 to 5 in which the metal alkoxide is an ethoxide, propoxide or a butoxide.
- 25 9. A structure which incorporates a layer of lithium quinolate and a means to pass an electric current through the lithium quinolate layer.
- 30 10. An electroluminescent device comprises a conductive substrate which acts as the anode, a lithium quinolate layer and a metal contact connected to the electroluminescent layer which acts as the cathode.

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11. An electroluminescent device as claimed in claim 10 in which the substrate is transparent conductive glass or plastic material.
- 5 12. An electroluminescent device as claimed in claim 11 in which there is a hole transporting layer deposited on the transparent substrate and the lithium quinolate is deposited on the hole transporting layer.
- 10 13. An electroluminescent device as claimed in claim 12 in which the hole transporting layer is made of a film of poly(vinylcarbazole), N,N'-diphenyl-N,N'-bis(3-methylphenyl)-I,I'-biphenyl -4,4'-diamine (TPD), polyaniline.
14. An electroluminescent device as claimed in any one of claims 10 to 12 in which a dye is included to modify the colour spectrum of the emitted light.
- 15 15. An electroluminescent device as claimed in any one of claims 10 to 14 in which the lithium quinolate is mixed with a polyolefin and the amount of lithium quinolate in the mixture is from 95% to 5% by weight of the mixture.
- 20 16. An electroluminescent device as claimed in claim 16 in which the amount of lithium quinolate is from 25 to 20% by weight of the mixture.
- 25 17. An electroluminescent device as claimed in any one of claims 12 to 16 in which a hole transporting material is mixed with the lithium quinolate in a ratio of 5-95% by weight of the lithium quinolate to 95 to 5% by weight of the hole transporting compound.
- 30 18. An electroluminescent device as claimed in any one of claims 12 to 17 in which there is a layer of an electron injecting material between the cathode and the lithium quinolate layer,

19. An electroluminescent device as claimed in any one of claims 12 to 17 in which there is an electron injecting layer mixed with the lithium quinolate
- 5 20. An electroluminescent device as claimed in claim 18 in which the electron injecting layer is a different metal quinolate which will transport electrons when an electric current is passed through it.
- 10 21. An electroluminescent device as claimed in claim 18 in which the electron injecting layer is aluminium quinolate
- 15 22. An electroluminescent device as claimed in claim 12 in which there is a substrate formed of a transparent conductive material which is the anode on which is successively deposited a hole transportation layer, the lithium quinolate layer and an electron transporting layer which is connected to a metal anode.

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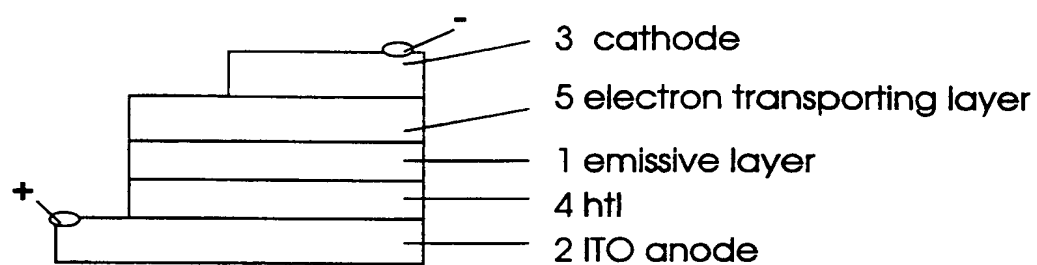


Fig. 1

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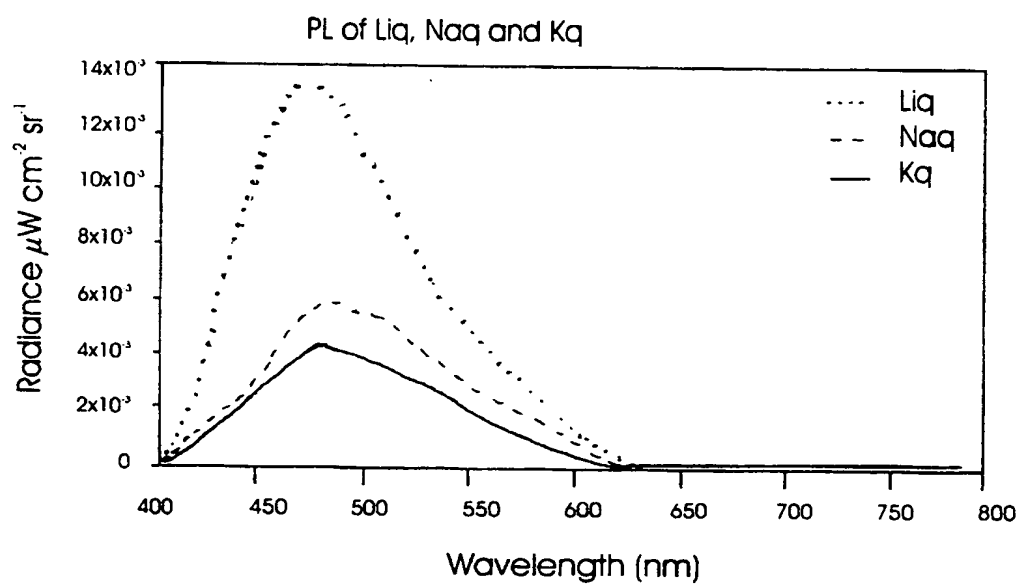


Fig. 2

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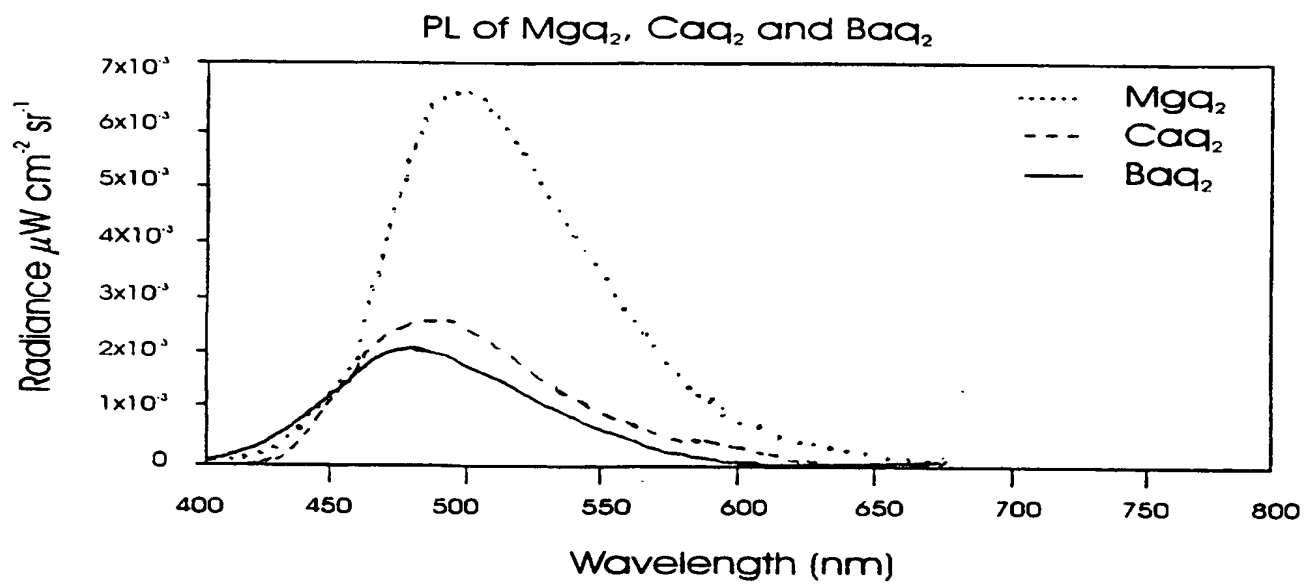


Fig. 3

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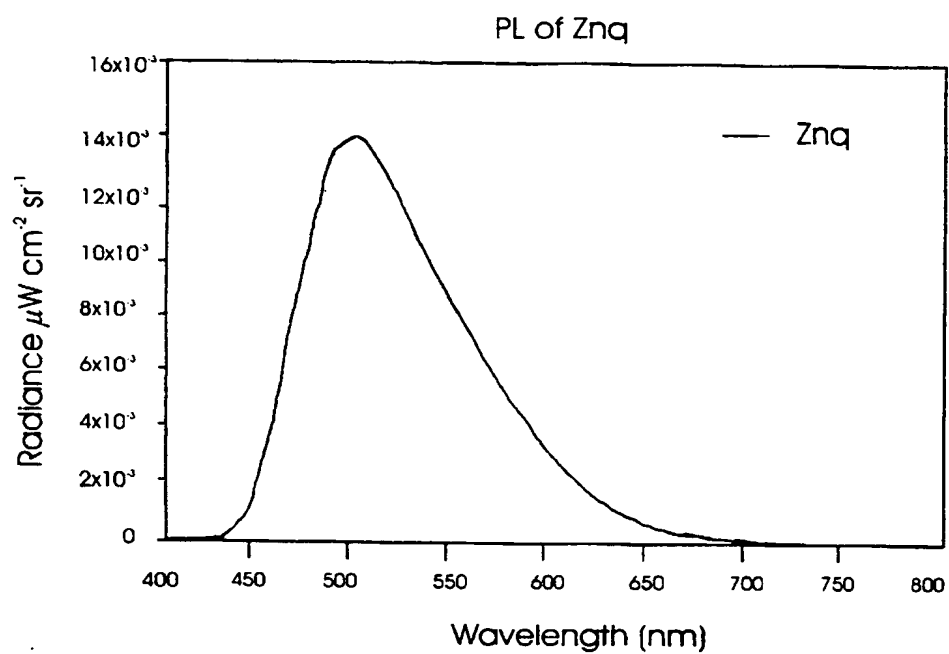


Fig. 4

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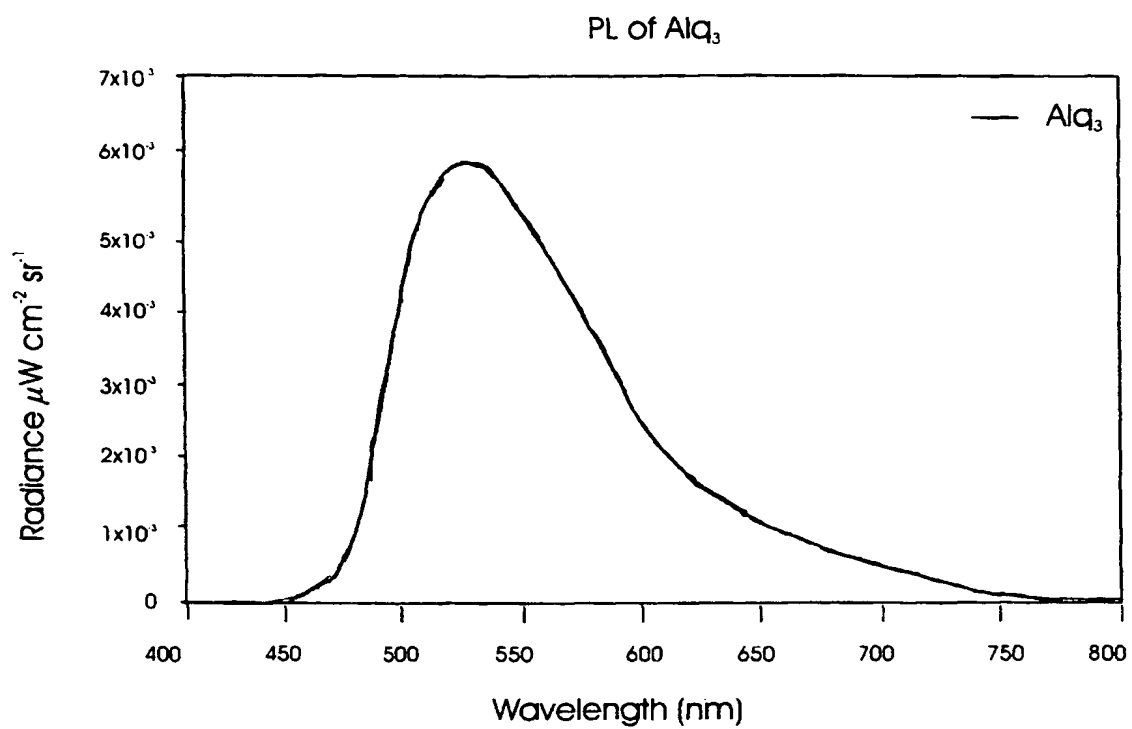


Fig. 5

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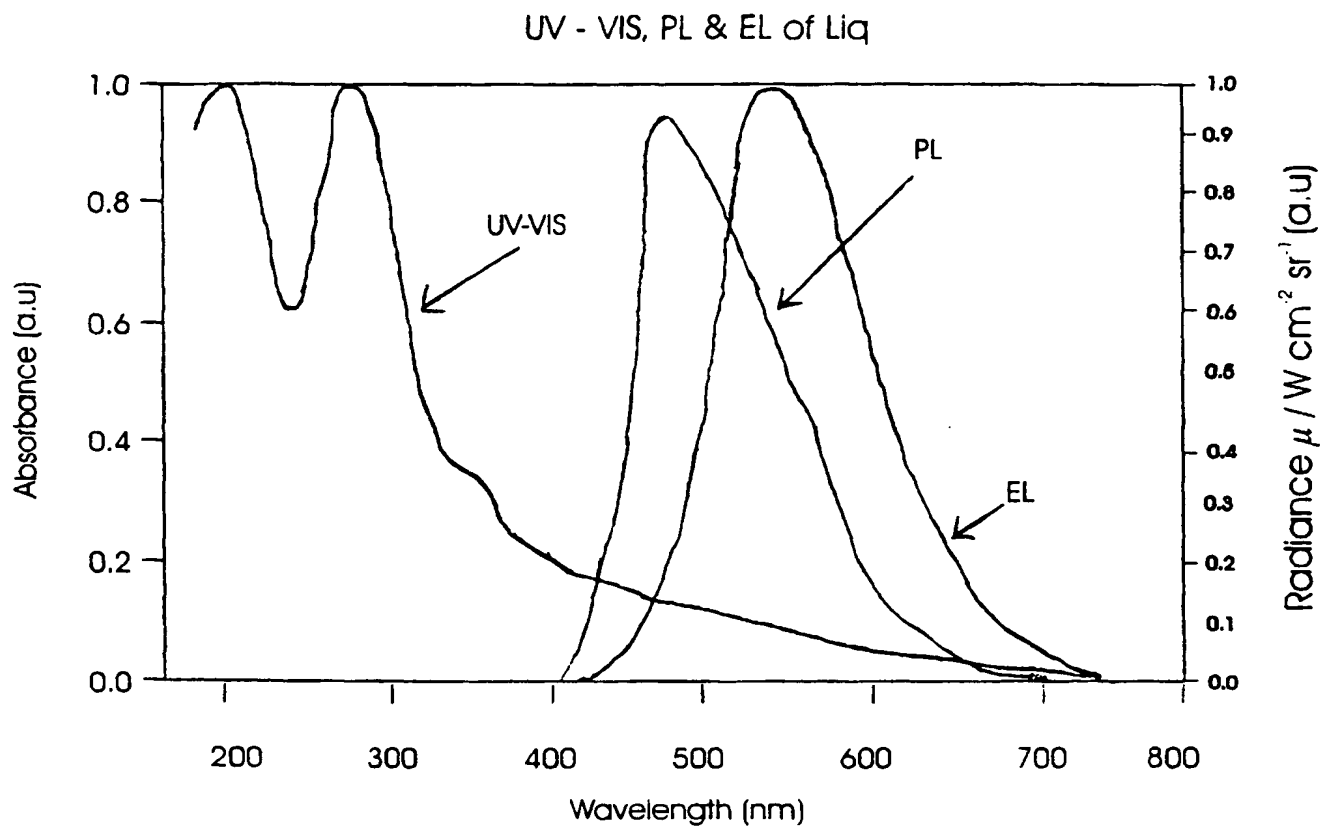


Fig. 6

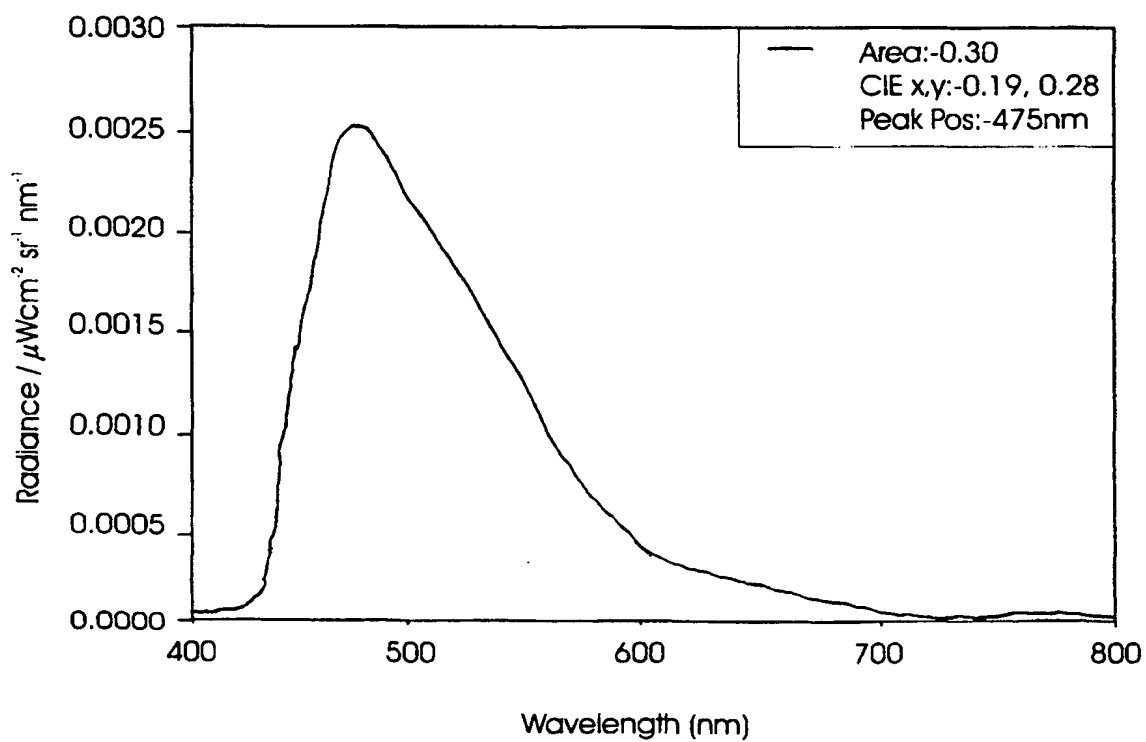
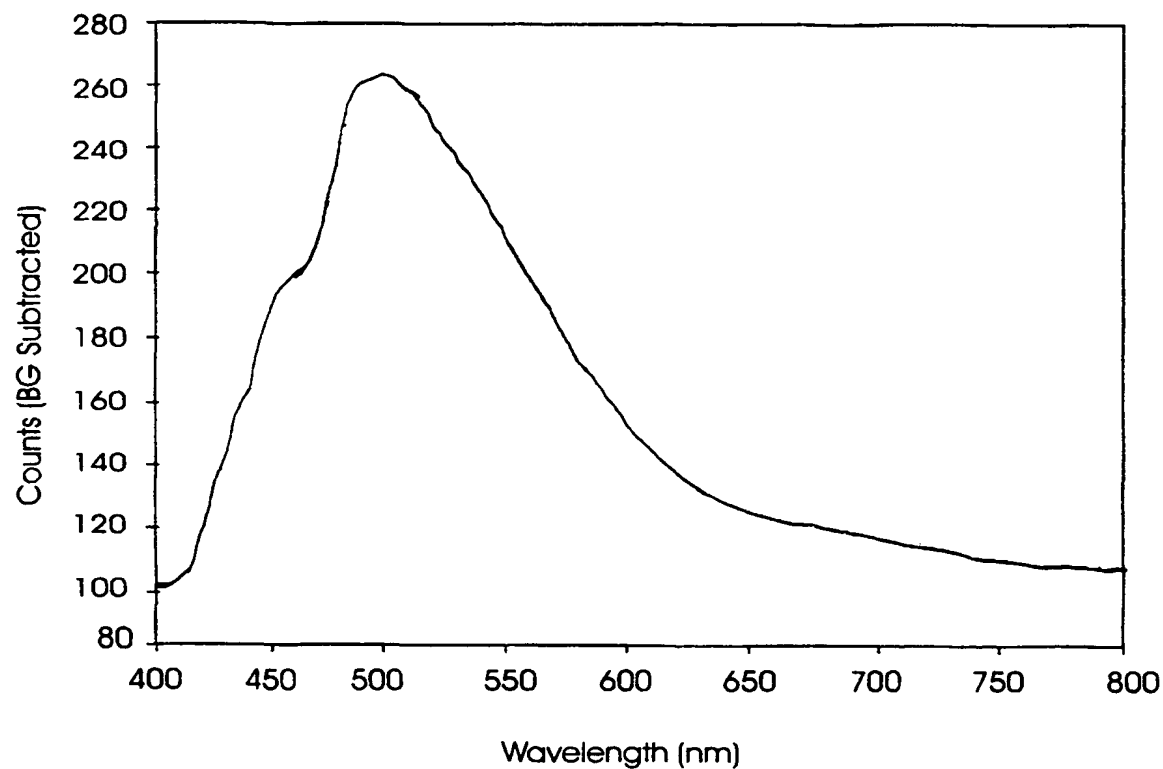


FIG. 7